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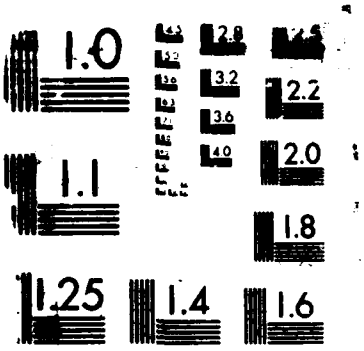

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The report describes the first year implementation of the Center for Excellence on Cement Paste Matrix Composite Materials. Topics include organization and administration, equipment acquisitions, personnel and the development of a comprehensive research program. Preliminary results on projects involving processing of MDF cements and the electrical properties of DSP cements are presented.

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CEMENT PASTE MATRIX COMPOSITE MATERIALS CENTER

First Annual Report
(29 October 1986 - 29 October 1987)

submitted to

U. S. Air Force Office of Scientific Research
Bolling Air Force Base
Washington D.C.

Prepared by: J. F. Young and R. L. Berger

October 1987
Department of Civil Engineering
University of Illinois at Urbana-Champaign
Urbana, IL 61801

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Summary

The implementation of the research program of the Cement Paste Matrix Composite Materials Center has proceeded well during its first year of operation. The Center has been designated as a distinct program, known as the Center for Cement Composite Materials, and is expected to be approved by the Board of Trustees soon. Remodelling of laboratory space was funded by the College of Engineering and office space allocated by the Department of Ceramic Engineering. Financial record keeping has been developed and refined; the Department of Civil Engineering is returning some overhead charges to the Center.

The majority of the itemized equipment has been acquired or is on order. About half of this is now in operation, the remainder being intstalled at the present time. A laboratory manager has been hired and operational procedures being developed.

Nine research projects have been identified and all had commenced by September 1987. Only two projects have been active for more than six months, however. Two presentations on Center-supported research have been given at technical meetings, and one is submitted for publication.

An Advisory Committee was appointed and met in July 1987 to review the Center's research program. Initial planning has begun for an Industry Affiliates Program.

1. Introduction

As had been anticipated, technical progress was limited during the first year of funding for several reasons:

1. The delay in approval of the contract with the Air Force and release of operating funds.
2. Acquisition of equipment.
3. The bulk of the first year funding being allocated to equipment.
4. The need to remodel laboratory space for Center activities.

Nevertheless sufficient progress was made to put the Center on a sound footing and by September 1987 all scheduled projects had been started. The support of the Departments of Civil Engineering and Ceramic Engineering and the College of Engineering was a critical factor in minimizing the impact of the start up delays. Temporary support of personnel and prompt scheduling of planned remodelling were instrumental in beginning some research in late 1986.

2. Organization and Administration

2.1 Center Organization

The Center has been set up as a separate unit which is administered by the Department of Civil Engineering. A formal proposal to this effect was prepared by the College of Engineering in 1987, reviewed by the Graduate College in 1987 and approved by the Campus Senate in October 1987. Approval by the Board of Trustees is expected in late 1987 or early 1988. The name has been shortened to **Center for Cement Composite Materials.**

The Department of Ceramic Engineering has provided an office suite for the Center to accommodate the Director and a secretary. The office is very favorably located in the Ceramics building and is geographically centered with respect to the Center's research activities.

2.2 Financial Administration

The financial affairs of the Center are being handled by the Business Office of the Department of Civil Engineering. A special computerized accounting system was developed to keep track of the individual research projects, each of which have been given a separate operating budget. A set of procedures for project management was developed and circulated to all faculty associates. Since the Center involves faculty and students from several different campus units keeping track of records is more difficult than for most research projects. agreement has been reached with the Department to share that part of the indirect costs generated by the Center that would normally accrue to the Department.

3. Research Program

During the first few months of the contract nine research projects were identified to meet the objectives of the Center. These are listed in Table 1. However, for reasons discussed earlier only a few of these were active during most of the year. Only two were started in October 1986, three more in June 1987, and the remaining four began in August 1987. Consequently there are few technical advances to report at this time. In addition two ongoing projects are partially supported by the Center in that the graduate students are assisting in setting up Center facilities. Both projects are related to the Center's objectives, although since they are directed more towards the long term objectives of the Center and not the short term goals they are not being fully funded. The projects are:

The Effect of Chemical Doping and Phase Transformations on Microstructural Development of Dicalcium Silicate

P.I.: J. F. Young; G. R. A.: C. J. Chan

The Influence of Staring Crystal Structures on the Formation High Surface Area Silica by by Carbonation

P.I.: R. L. Berger; G. R. A.: J. M. Bukowski

Table 1 - Summary of Research Projects

Project #	Title of Center Project	Principal Investigators	Faculty Associates	Research Assistants	Start Date	Project Funding*
21	MDF Cement Processing	M. Berg	R. Wool J. F. Young	P. Russell	10/86	$49,583
22	Electrical Properties of Cements	D. A. Payne J. F. Young		D. Leigh	10/86	$50,179
23	Fiber/Matrix Adherence	J. Homeny S. D. Brown		D. Hansen	6/87	$37,295
24	Intercalation of Polymers in Inorganic Crystals	S. I. Stupp		J. Hansen P. Messersmith	8/87	$37,868
25	Polymer-Solid Interactions	S. Granick		T. C. McGlinn	8/87	$34,067
26	Polymer-Cement Interfaces	R. P. Wool		H. Zhang	8/87	$38,734
27	Phosphate-Bonded Cements	W. G. Klemperer	J. F. Young	L. Babcock S. P. Lockledge S. Yang	6/87	$30,171
28	High Strength Cement Pastes	R. L. Berger J. F. Young		J. Shunkwiler	8/87	$30,949
29	Setting Processes for Cements	C. F. Zukoski		L. B. Chen	6/87	$34,901

*Until 8/20/87

3.1 Current Technical Progress

Sufficient progress has been made only on projects #21 and #22 to warrant a detailed account of progress.

Project #21 - Processing of MDF (Macro-Defect Free) cement was started on a small laboratory 3-roll mill using the formulation published by Imperial Chemical Industries (ICI). By May 1987 the procedures had been refined to the point at which a good quality material could be produced with flexural strength equal to, or better than, the values reported by ICI. (Table 2). A switch was made in late July to a larger 2-roll mill with greater control of mill parameters. This required a renewed optimization of the process. Some initial experiments have been made to chemically modify the material to reduce water sensitivity.

Table 2
Initial Properties of MDF Cements

Property	This work	Reported by ICI
Bulk Density (g/cm^3)	2.42 ± 0.05	<2.5
Flexural strengths (ksi)	30 ± 2	25
Youngs Modulus (10^6 psi)	8.2 ± 0.7	7.3
Shear Modulus (10^6 psi)	3.2 ± 0.3	3.0
Poisson's Ratio	0.29 ± 0.01	0.2

Project #22 - This project has been studying the electrical properties of DSP (Densified with Small Particles) formulations compacted under uniaxial pressure of 10,000 psi, and hydrated at room temperature for several days. After strong drying to remove excess water the compacts show properties equal to, or better than sintered alumina (Table 3). However exposure to water vapor, even at low humidities severely degrades these properties. Studies are underway to reduce the influence of water on these properties.

Table 3

Electrical Properties of DSP Cements

Exposure (% r.h)	Resitivity (ohm.cm)	Dielectric Constant	Dielectric Loss (Tan γ, %)
0	10^{13}	5	0.6
11	10^{9}	7	13.3
50	10^{8}	18	18.5
100	10^{7}	22	29.3

3.2 Research Presentations

The following research presentations were given during the year:

1. J. M. Bukowski and R. L. Berger, "Thermally Evolved Gas Analysis of Carbonated Calcium Silicates". Presented at the Annual Meeting of the North American Thermal Analysis Society, Washington D.C. Sept. 1987

2. D. W. Leigh, D. A. Payne and J. F. Young, "Preparation and Properties of DSP Cements for Electrical Applications", Ceramic Substrates and Packages - an International Symposium, Denver, CO, October 1987.

No papers were published during the year, but presentation No. 2 has been submitted for the Proceedings volume.

4. Equipment

During the first year $439,000 was budgeted for acquiring major items of equipment. The bulk of the instruments has now been ordered and installed, and this is described in Table 4. All are state-of-the-art instruments and in most cases automated, being controlled by a personal computer or other microprocessor. A major factor in selection of these instruments was that they would be used by many investigators.

In addition a number of items costing less than $10,000 have been acquired, and are listed below

25-ton Hydraulic press with heated platens (Carver & Sons)		$6,678
Forced convection oven (Despatch Industries)		$2,660
Noise Filter for PGT, System 4 (Topaz Inc)		$1,195
Sputter Coater, Model IB-3 (for SEM) (RMC Industries)		$3,975
pH meter & electrodes	Orion	$2,235
Analytical & Laboratory balances (2)	Mettler	$3,592
Back-scatter detector	Model 113A (for SEM) GW Electronics	$7,135

Table 4

Status of Major Equipment Purchases

Instrument	Supplier & Model	Cost	Status
2 roll mill	Stannant (Used model)	$32,000	In operation
Mercury Porosimeter	Micromeritics, Model 9220 (2 stations)	$33,890	In operation
Nitrogen Adsorption	Micromeritics, Model ASAP 2400 (6 stations)	$64,213	In operation
X-ray/Image Analysis System (for SEM) interface	Princeton-Gamma-Tech, System 4	$52,280	In operation, but some problems
X-ray diffractometer	Rigaku, Model Geigerflex Dmax-II	$101,815	Being installed
Particle Classifier	American Alpine Model 100 MZR	$32,890	Received, but not yet operating
Small strain Creep Apparatus	Bohlin Rheologi	$56,000	On order
Particle Size Analyzer			Being evaluated
UV-VIS Spectro-photometer			Being evaluated
Contribution to an FTIR spectrometer (to be used for Project #25	Nicolet, IR-32	$11,700	On Order

5. Personnel

There are 13 faculty associates assisting the Principle Investigators in directing the research projects funded by the center. They are listed in Table 5. Only nine of the thirteen are actively involved in directing research; of the four inactive associates two joined the Center during 1987.

Table 6 shows the Graduate Research Assistants who have been appointed to work on the research projects. In addition J. M. Bukowski and C.-J. Chan, Ph.D. candidates in Ceramics are assisting in instrument acquisition and student training.

Mr. Thomas Bier was hired as a research associate in September 1987 to oversee the laboratories: maintain the instruments and train students to use them. Mr. Bier was selected, after the position was advertised, from a list of 12 candidates. Mr. Bier will receive his Ph.D. in Civil Engineering - Materials from the University of Karlsruhe in December.

Table 5

Listing of Faculty Associates

Name	Departmental Affiliation	Research Interests
M. Berg*	MSE+-Ceramics	Ceramic Processing
S. D. Brown*	MSE+-Ceramics	Fracture testing
R. C. Buchanan	MSE+-Ceramics	Alumina phosphate cements
S. Granick*	MSE+-Ceramics	Polymer-solid interfaces
J. Homeny*	MSE+-Ceramics	Fracture of composites
R. J. Kirkpatrick	Geology	Mineral structures
G. Kordas#	MSE+-Ceramics	Electron paramagnetic resonance
W. G. Klemperer*	Chemistry	Synthesis of metal-oxygen structures
D. A. Payne*	MSE+-Ceramics	Electrical properties Ceramic processing
S. I. Stupp*	MSE+-Ceramics	Polymer-ceramic interfaces
C. L. Tucker	Mechanical Engineering	Polymer processing
R. Wool*	Materials Science	Polymer interfaces
C. F. Zukoski*	Chemical Engineering	Properties of colloids

*P.I. or Co-P.I. of a Center Project
+Material Science and Engineering
#Joined during 1987

Table 6

Graduate Research Assistants Employed to work on Projects

Name	Project #	%time	Date Appointed	Last Degree, & Institution & Date	Degree Sought
P. Russell	21	50	10/29/86	B.S., Ceramic Engr. U. Missouri-Rolla, 1979	M.S.
D. Leigh	22	50	10/29/86	B.S., Ceramic Engr. U. Illinois, 1986	M.S.
D. Hansen	23	50	5/20/87	B.S., Ceramic Engr. U. Illinois, 1987	M.S.
J. Hanson	24	25	8/20/87	B.S., Bioengr. U. Illinois, 1987	M.S.
P. Messersmith	24	25	8/20/87	B.S., Bioengr. Clemson, U. 1987	B.S.
T. C. McGlinn	25	50	8/20/87	Ph.D., Physics U. Illinois, 1987	
H. Zhang	26	50	8/20/87		Ph.D.
S. P. Lockledge	27	25	5/20/87	B.S., Chemistry Penn State	Ph.D.
S. Yang	27	25	5/20/87	B.S., Chemistry Nanjiang U.	Ph.D.
J. Shunkwiler	28	50	8/20/87	B.S. Ceramics Ohio State U., 1987	M.S.
L. B. Chen	29	50	5/20/87	B.S. Chem. Engr. U. Washington, 1986	Ph.D.

6. Facilities

Considerable space was provided by the Department of Ceramic Engineering for the Center activities. This included an office for the Director and a secretary, two laboratories, and a small room for the air classifier. One laboratory and the room were extensively remodelled with funds provided by the College of Engineering. Instrumentation for the characterization of powders and porous materials is being assembled in the laboratory and will be administered by the Center as a joint facility with the Department. Modest user fees are charged to cover operating costs and maintainance. Center projects are given priority access and charged lower fees.

Additional space is being provided by the Department of Civil Engineering. A room was remodelled with College funds to accommodate a scanning electron microscope being purchased by the Center for Advanced Construction Technology, but administered by the Center under similar arrangements as above.

7. Other Activities

7.1 Advisory Committee

An advisory committee was appointed in consultation with Major Hager to review and comment on Center activities. the members are listed in Table 7. Members are appointed through the end of 1988, but we expect Major Hager to be replaced by the Program Manager at AFOSR overseeing the Center.

A meeting was held at the University of Illinois in July 1987 at which time presentations of the planned research program were made. The program was favorably received. The committee recommended a two-day meeting should be held in the Fall of 1988 and this recommendation has been accepted.

7.2 Industry Programs

Considerable interest has been shown by industry following the announcement of the Center. After extended discussions with the College of Engineering a tentative decision has been made to develop an Industry Affiliates Program in 1988. The College will fund the cost of the initial meeting and supply the necessary support for its organization.

The Weyerhausser Foundation has indicated that it will donate $12,500 to the Center before the end of the year as an unrestricted donation to further the Center's programs. Mr. Paul Russell was awarded $6,000 from the International Society of Hybrid Microelectronic Foundation to study the use of MDF cements as a possible substrate material. Several companies have donated materials for use in the research programs.

Table 7

Advisory Committee Members

Name	Affiliation	Location
Major J. W. Hager (Chairman)	Electronic & Mater. Sci. UFOSR	Bolling AFB, DC
Prof. S. Diamond	School of Civil Engr. Purdue University	West Lafayette, IN
Dr. G. J. C. Fohnsdorff	Div. of Bldg. Materials N.B.S.	Gaithersburg, MD
Captain C. Felice	Air Force Weapons Lab	Kirtland AFB, NM
Mr. J. Hayes	Air Force Engineering Services Center	Tyndall AFB, FL
Mr. A. Koblin	CEMCOM Research Associates	Lanham, MD
Mr. C. S. Saunders	Composite Mater. Group ICI Advanced Materials	Washington, DC

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